Synthesis and Application of Breadfruit Peel-Based Magnetic Activated Carbon for Pb(II) Removal from Electroplating Wastewater

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Abstract

The electroplating industry is one of the producers of liquid waste containing lead heavy metal (Pb(II)) which is a high risk to the environment and human health. Pb(II) metal is a toxic heavy metal, does not decompose easily, is bioaccumulative, and its solubility is strongly influenced by pH. In acidic conditions, Pb is more easily soluble as Pb^{2+} ions, in alkaline conditions it can form deposits such as $Pb(OH)_2$. Based on its toxic and persistent nature, Pb needs to be treated before wastewater is discharged into the environment. This study aims to process electroplating liquid waste through the development of magnetic activated carbon based on breadfruit bark synthesized using the precipitation method, as well as to examine the effectiveness of its application in the removal of Pb(II) ions from electroplating liquid waste. The characterization of magnetic activated carbon as an adsorbent is carried out using various analysis techniques to determine the surface morphology, pore structure, and specific surface area. Adsorption tests were performed to evaluate the effect of contact time, and adsorbent dose on Pb(II) removal. The results showed that magnetic activated carbon has a high ability to absorb Pb(II), with a removal of 96.79%. the optimal conditions were obtained at an adsorbent mass of 3.424 g and a contact time of 79.137 minutes, with a prediction of Pb(II) removal of 98.083%

Keywords: breadfruit bark, magnetic activated carbon, adsorption, lead, electroplating waste water

1. Introduction

Industrial waste watercontaining heavy metals has become one of the sources of environmental pollution that is now a global concern. Pb(II) metal in electroplating wastewater is a serious problem for ecosystems and human health because it is difficult to degrade naturally. The hazards posed by lead in electroplating waste waterlead to the need to develop effective, economical, and sustainable technologies to remove lead from wastewater. Several methods, including chemical precipitation, coagulation-flocculation, adsorption, ion exchange, membrane filtration, electrochemical processes, and phytoremediation, can be used to treat waste watercontaining Pb(II) metal. Activated carbon adsorption method has been developed to a great extent, due to its high removal efficiency, simple operation process and potential application of cheap and environmentally friendly raw materials. The activated carbon adsorption method has been extensively studied due to its high removal efficiency, simple operation and the possibility of using cheap and environmentally friendly raw materials. [1]

Various studies have developed the use of biomass as a raw material for making activated carbon because of its abundant availability, relatively low cost, and in line with sustainability principles. Activated carbon produced from olive seeds and almond shells, carbonized at 600 °C and activated with KOH, H_3PO_4 and $ZnCl_2$ at 850 °C, can remove up to 100% of Pb(II) at an initial concentration of 500 mg/L and an adsorbent dose of 5 g/L. (Rahimi et al., 2025). Moreover, the modified sugarcane bagasse-based activated carbon prepared using nitric acid (HNO3) oxidation exhibited an improved ability to adsorb Pb(II). The characterization results indicated that the HNO3 modification reduced the specific surface area because of the micropore blockage, but increased the number of oxygen-containing functional groups such as hydroxyl, carboxyl, carbonyl and esters on the surface of the adsorbent.[2]. The significant increase of functional groups plays an important role in Pb(II) adsorption, and its maximum adsorption capacity is calculated to be 212.31 mg/g according to the Langmuir model.[3]

The latest technological developments in the manufacture of adsorbents currently lead to the modification of activated carbon with magnetic substances into magnetic activated carbon (MAC). This modification is generally achieved by adding magnetic particles, such as Fe_3O_4, to the surface or

structure of activated carbon, so that the adsorbent not only has good adsorption capacity but can also be quickly separated from the solution using an external magnetic field.[4]. This technology is considered important because one of the disadvantages of conventional activated carbon is the difficulty of separation and reuse after adsorption. Magnetic activated carbon results from co-pyrolysis of lignite and poplar leaves for the adsorption of Pb(II) and Cd(II) ions in wastewater. The resulting MAC contains Fe_3O_4, has a saturated magnetization of 13.83 emu/g, a specific surface area of 805.86 m^2/g, and a micropore volume of 0.23 cm^3/g. At optimal pH 5, contact time of 120 minutes, adsorbent dose of 4 g/L, and temperature of 30 °C, MAC can separate Pb(II) and Cd(II) by 84.40% and 78.80%, respectively; in addition, these adsorbents can be separated from the solution using a magnetic field and still maintain an adsorption capacity of Pb(II) of 84.90%. (Zhang et al., 2022).[5]

In the development of quality magnetic activated carbon must be through characterization tests and performance tests. Physical and chemical characterization tests were performed to characterize the adsorbent. The surface morphology was characterized by scanning electron microscopy (SEM), the metal element content was determined by SEM-EDX, the specific surface area was determined by the Brunauer–Emmett–Teller (BET) method, and the surface functional groups were identified by Fourier transform infrared spectroscopy (FTIR). Meanwhile, the performance tests are conducted to determine the adsorbent's adsorption capacity and maximum adsorption capacity for metal ions in waste water .[5]. This research aims to develop magnetic activated carbon based on breadfruit peel waste as a Pb(II) adsorbent in waste water . The resulting magnetic activated carbon is studied based on its surface morphology before and after the magnetization process. In addition, this study evaluated the effect of adsorbent dose on the percentage of Pb(II) removed from electroplating wastewater. It determined the optimal adsorption conditions for variations in adsorbent and contact time.

2. Method

2.1 Raw Materials

The main raw material used in this study is breadfruit peel (*Artocarpus altilis*) obtained from small industrial waste processing of breadfruit. The chemicals used include iron(III) hydrochloride hexahydrate (FeCl3·6H2O), iron(II) sulfate heptahydrate (FeSO4·7H2O), sodium hydroxide (NaOH), hydrochloric acid (HCl), potassium dichromate (K2Cr2O7), and aquades. Samples of electroplating waste obtained from the metal plating industry in Surabaya, Indonesia

2.2 Preparation of breadfruit peel

The skin of breadfruit is washed using running water to remove dirt and other impurities, then rinsed with aquades. The sample is dried in the oven at 105°C for 24 hours until the moisture content is constant. After drying, the sample is ground in a grinder and sifted to a particle size of 60–100 mesh.

2.3 Magnetic Activated Carbon Synthesis and Activation

2.3.1 Manufacture of activated carbon of breadfruit peel

The carbonization process is carried out on breadfruit skins that have been dried using a furnace. The resulting breadfruit skin carbon is then reduced in size until a size of 200 mesh is obtained. The activation process is carried out through chemical activation and physical activation. Chemical activation is performed by soaking the carbon in a 1 M H_3PO_4 solution for 8 hours, followed by filtration and oven drying. Physical activation is performed after chemical activation using a microwave at 450 watts. During the physical activation process, N_2 gas is flowed into the microwave to keep the microwave environment air-free. The resulting activated carbon is then analyzed using SEM-EDX and surface area assays.

2.3.2 Manufacture of magnetic activated carbon of breadfruit peel

The manufacture of magnetically activated carbon begins with the preparation of an Fe3O4 solution, obtained by mixing FeCl3 and FeSO4 solutions at a molar ratio of 2:1. Furthermore, the resulting Fe3O4 is mixed with activated carbon that has been stirred in 200 mL of water while heating. In the mixing of Fe3O4 solution with activated carbon, it is carried out in a variation of the mass ratio of activated carbon (AC) and iron oxide / Fe3O4 (FO) namely (AC: FO = 2:1); (AC: FO = 3:2) ; (AC: FO = 1:1) ; (AC: FO = 2:3); and (AC: FO = 1:2), to each mixture is added 100 mL of NaOH solution

2.5 M drops by drop until it runs out with a *stirring time* of 3 hours. The formed carbon magnetite is separated from the solution by filter paper. The obtained carbon magnetite is washed several times with water until the conductivity value of the filtered water is < 100 µS/cm2. The carbon magnetite is further dried in the oven at 110 °C for 2 hours, then mashed and sifted through a 100-mesh sieve. Next, the characterization of activated carbon and magnetic activated carbon was carried out.

2.3.3 Absorption of chrome in waste waterusing magnetic activated carbon

Electroplating waste treatment is carried out by the batch adsorption method. The volume of waste used is 50 ml, while the magnetic activated carbon dose is 2; 4; 6; 8; and 10%.The adsorption time variations used were 60, 90, 120, and 180 minutes. The results obtained from the waste treatment process were then analyzed for chromium content and calculated chrome removal, and the effect of magnetic activated carbon dose and adsorption time on chromium removal.

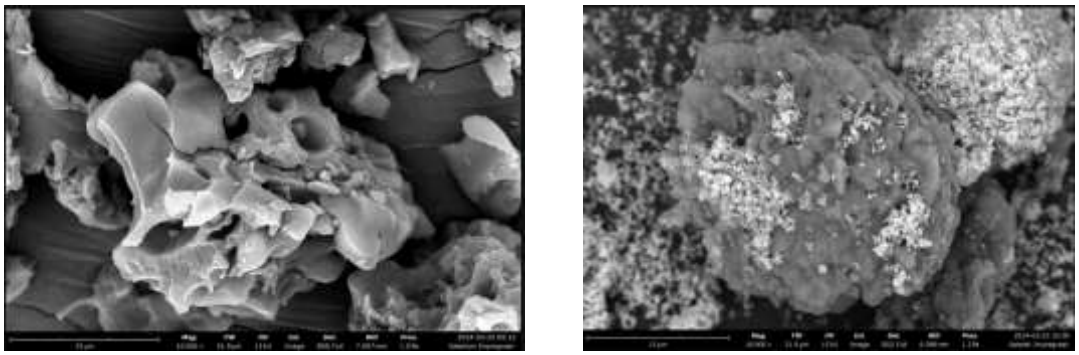
Calculation of chromium removal using the equation:

$$\%\, removal = \left(\frac{Ca - Co}{ca}\right) \text{x} 100\%$$

Where Ca is the initial Concentration (Mg/l); Co is the final concentration (Mg/l)

3. Results

Surface Morphology



(a) (b)

Figure 1. Surface morphology of activated carbon of breadfruit bark:
(a) before magnetic modification and (b) after magnetic modification.

The surface morphology of activated carbon before and after magnetization was examined by scanning electron microscope (SEM) in Figure 1. The structure of the activated carbon before the magnetization process is irregular, rough and flake-like (Figure 1 (a)). Moreover, cavitation, crevices and fractured surfaces are observed which indicate that the carbonization and activation process have decomposed organic components in breadfruit bark and produced a more open form of carbon. This rough and porous surface morphology plays an important role in providing contact areas and active sites for the Pb(II) ion adsorption process.

After the process of magnetic modification, as shown in Figure 1(b), there is a fairly pronounced change in surface morphology. The surface of activated carbon appears more heterogeneous and is covered by small, lighter-colored particles scattered on the surface of the adsorbent. The particles are thought to be Fe_3O_4 particles that are formed during the process of coprecipitation and are deposited on the surface of activated carbon. The presence of these particles indicates that the magnetic modification process was successfully carried out. This is in line with the research by Zhang et al. (2022), who demonstrated the successful formation *of magnetically activated carbon by* the presence of Fe_3O_4 particles and the distribution of Fe on the surface of the activated carbon.[5]

However, Figure 1(b) also shows the agglomeration of magnetic particles on several parts of the carbon surface. Fe_3O_4 agglomeration can cause partial pore closure, potentially reducing the active surface area and accessibility of Pb(II) ions to the adsorption site. This condition suggests that the

distribution of magnetic particles should be controlled to prevent magnetic modifications from significantly degrading adsorption performance. However, the presence of Fe_3O_4 particles provides an additional advantage: they facilitate separating the adsorbent from the solution after adsorption using an external magnetic field.[5]

Thus, the morphological changes after magnetization indicate that the activated carbon from breadfruit husks has been successfully modified into magnetic activated carbon. The rough, heterogeneous surface characteristics and the presence of magnetic particles on the surface of the adsorbent indicate that this material has the potential to be used as a Pb(II) adsorbent in the treatment of electroplating waste water .[6]

Effect of magnetic activated carbon adsorbent dose on Pb removal %

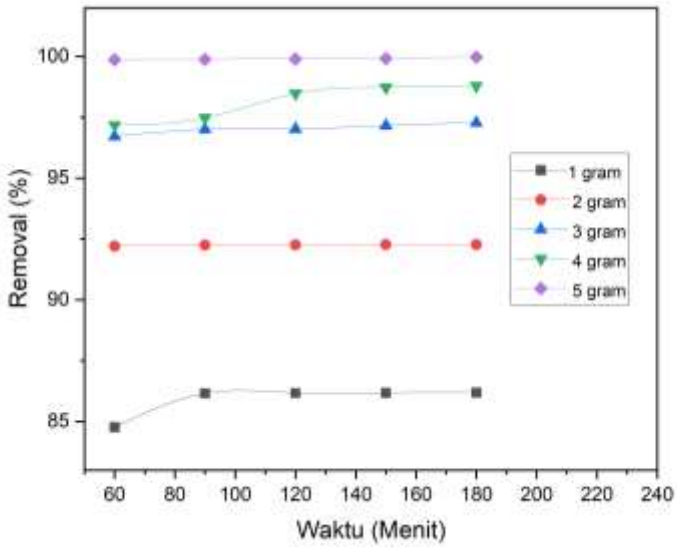


Figure 2. Effect of magnetic activated carbon adsorbent dose on Pb removal %

The effect of magnetic activated carbon adsorbent dose on the percentage of Pb(II) removal is shown in Figure 2. Based on Figure 2, adsorbent doses from 1 gram to 5 grams lead to increased Pb(II removal. At a dose of 1 gram, the removal of Pb(II) is in the range of about 84–86%. When the adsorbent dose is increased to 2 grams, removal increases to about 92%. Furthermore, at doses of 3 grams and 4 grams, Pb(II) removal increased to about 96–97% and 97–99%, respectively. The highest removal is achieved at a dose of 5 grams, approaching 100%.

The increase in Pb(II) removal efficiency as the adsorbent dose increases is due to the increase in the number of active sites and the available contact surface area of the adsorbent. The greater the mass of magnetic activated carbon used, the more pores and surface-active groups are available to interact with Pb(II) ions, thus the greater the chance of adsorption. This phenomenon is in line with previous research that increased adsorbent doses can increase Pb(II) elimination due to an increase in the number of active sites on the adsorbent surface.

In addition to the adsorbent dose, the contact time also affects the adsorption process. The graph shows that the increase in contact time from 60 to 180 minutes provides only a relatively small increase in efficiency, especially at doses of 2 grams, 3 grams, and 5 grams. This indicates that the adsorption process is fast in the early stages because many active sites remain available. Once most of the active sites are occupied by Pb(II) ions, the adsorption rate decreases, and the system approaches equilibrium. Pb(II) adsorption studies using modified activated carbon also reported that the Pb(II) adsorption process can be rapid and follow a pseudo-second-order kinetic model, which shows the involvement of chemical interactions such as ion exchange and surface complexation.

At a dose of 1 gram, an increase in contact time from 60 minutes to 90 minutes increases the removal of Pb(II), but after that it tends to be stable. This suggests that at low doses, the number of active sites is relatively limited, so the adsorbents reach saturation more quickly. At a dose of 4 grams, the increase

in contact time still increases efficiency to about 120–150 minutes, then tends to be nearly stable. Meanwhile, at a dose of 5 grams, Pb(II) removal is already very high after 60 minutes and remains close to 100% for up to 180 minutes. This indicates that a dose of 5 grams provides a sufficient number of active sites to adsorb almost all of the Pb(II) ions in the solution.

The characteristics of magnetic activated carbon also support the high Pb(II) elimination performance in this study. Activated carbon has a porous structure, a high surface area, and surface functional groups that can play a role in the binding of heavy metal ions. In magnetic activated carbon, the presence of Fe_3O_4 not only provides magnetic properties, but can also contribute to adsorption interactions through electrostatic forces, surface complexation, ion exchange, and surface precipitation. Research on magnetic activated carbon for Pb(II) and Cd(II) adsorption shows that the adsorption performance of heavy metals is influenced by specific surface area, pore structure, surface functional groups, as well as electrostatic and complexation mechanisms. [7]

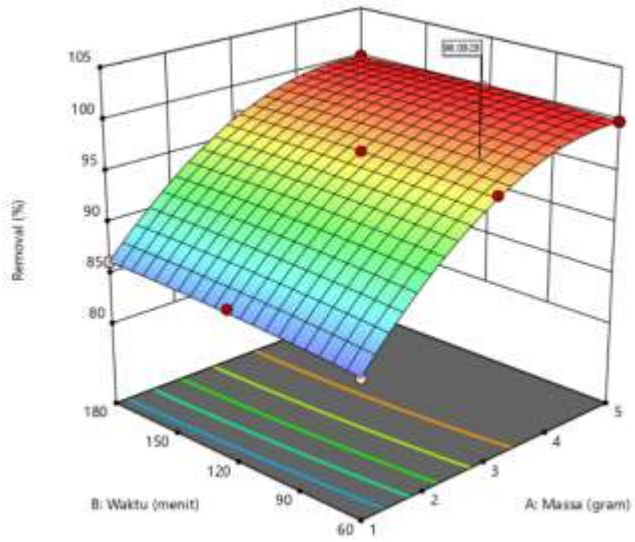
These results are in line with the research of Kiani Zadeh and Jafari, who used activated carbon/alginate/Fe_3O_4 magnetic composites for Pb(II) elimination. The study confirmed that magnetic activated carbon-based materials are effectively used as Pb(II) adsorbents, with characterization by SEM, EDX, FTIR, XRD, BET, and VSM demonstrating the successful formation of magnetic adsorbents. In addition, Zhang et al. stated that modifying activated carbon into magnetic activated carbon is an effective strategy to maintain adsorption capacity while facilitating separation of the adsorbent from the solution after adsorption using a magnetic field.[8]

Overall, the results of this study show that adsorbent dosage is an important factor in the elimination of Pb(II). The dose of 5 grams provides the highest removal, which is almost 100%. However, in terms of adsorbent efficiency, a dose of 4 grams at a contact time of 120–150 minutes is also effective, as it has achieved very high Pb(II) removal. Thus, magnetic activated carbon has the potential to be used as an effective adsorbent to lower Pb(II) levels in waste water .

Pb(II) Removal Optimization

Optimization was performed to determine the best conditions between the mass of the magnetic activated carbon adsorbent and the contact time for the percentage of Pb(II elimination. Based on the optimization results shown in Figure 3, the optimal conditions were obtained at an adsorbent mass of 3.424 g and a contact time of 79.137 minutes, with a predicted Pb(II) removal of 98.083% and a desirability value of 1.000. These values indicate that the combination of adsorbent mass and contact time is the most effective condition for achieving high Pb(II) removal while maximizing adsorbent efficiency.

In general, an increase in adsorbent mass increases the percentage of Pb(II) removal due to the increase in the number of active sites, contact surface area, and pores available to bind Pb(II) ions. Although a dose of 5 g results in removal close to 100%, the condition is not selected as optimal because the increase in removal is relatively small compared to the additional mass of adsorbents used. The effect of contact time is relatively smaller than that of adsorbent mass, suggesting that the adsorption process is rapid in the early stages and then approaches equilibrium.



Gambar 3. Response surface plot effect of mass of magnetic activated carbon adsorbent and contact time on Pb(II) removal

The optimization model showed excellent quality, as indicated by the R^2 value of 0.9994, Adjusted R^2 of 0.9992, Predicted R^2 of 0.9984, C.V. of 0.1680%, and Adeq Precision of 170.0687. These values indicate that the model has excellent suitability, accuracy, and predictive ability to explain the Pb(II) removal process.

4. Conclusion

The results showed that breadfruit peel-based magnetic activated carbon was effective for Pb(II) removal from electroplating wastewater, with the best experimental removal efficiency of 96.79%. Based on the optimization results, the optimum condition was achieved at an adsorbent mass of 3.424 g and a contact time of 79.137 min, with a predicted Pb(II) removal of 98.083%. Therefore, the prepared magnetic activated carbon has strong potential as an efficient adsorbent for Pb(II)-contaminated wastewater treatment.

Acknowledgments

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